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THESIS

**AN APPARATUS FOR MEASUREMENT
OF IGNITION AND BURNING CHARACTERISTICS
OF METALLIC PARTICLES**

by

Stephen Gulakowski

September, 1993

Thesis Advisor:

David W. Netzer

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**An Apparatus for Measurement of Ignition and Burning
Characteristics of Metallic Particles**

by

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Lieutenant, United States Navy
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Submitted in partial fulfillment
of the requirements for the degree of

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from the

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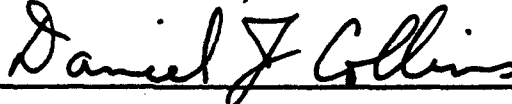
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ABSTRACT

An apparatus was designed for use in determining ignition and combustion characteristics of metallic fuel particles and liquid fuel droplets. The design was tailored around the available data acquisition systems and diagnostic instruments. The apparatus consisted of a small windowed combustion bomb, a CO₂ laser for particle ignition, a thermal imaging camera for recording the ignition process, a helium-neon laser and a 1024 element linear diode array sampled at 1Mhz for measuring forward scattered light and determining particle size (Fraunhofer diffraction). All major components were individually utilized. However, equipment limitations prevented actual use of the system for measurement of the burning rates of particles. Recommendations are made which would permit the original objectives to be met.

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I. INTRODUCTION

The addition of metallic particles to solid propellant rocket motors and ramjets has many well known advantages. They increase the energy output of the motor due to the high volumetric heating values of these metals compared to standard hydrocarbon fuels. The volumetric heating values for boron, aluminum, and JP5 are 32,200, 20,000, and 8230 cal/cm³ respectively [Ref. 1].

In airbreathing applications, these metallic fuels can be combined with hydrocarbon fuels to produce either slurry or solid fuels. The main disadvantage of the addition of metallic particles is generally a decrease in combustion efficiency. The residence times of the fuel particles in the combustor are often not long enough for complete combustion, therefore the combustion efficiency decreases. Considerable emphasis has been placed on enhanced burning rates of metallic fuel particles [Ref. 2].

The physical properties of metals and their oxides contribute greatly to a very unique combustion behavior. Table 1 contains thermodynamic data for selected materials [Ref. 2,3]. Metallic particles tend to accumulate on the burning fuel/propellant surface and then detach from the surface and burn in the combustor cavity. If a metal is molten at temperatures present on the burning surface, a

TABLE 1. THERMODYNAMIC DATA OF VARIOUS MATERIALS [Ref. 2,3].

Substance	T _{melt} [K]	T _{boil} [K]	Molecular Weight
B	2450	3931	10.8
Ti	1933	3591	47.9
TiB ₂	3193	-	69.5
TiO ₂	2143	3200	79.9
B ₂ O ₃	723	2316	69.78
Al	933	2750	26.98
Al ₂ O ₃	2320	3253	101.96

possibility exists that the smaller particles will combine to form larger particles which may have a longer burning time. The formation of a metal oxide layer on the surface of the particle slows down the ignition time and burning rate of the particle. If this surface oxide layer has a high melting temperature it may be difficult to ignite. [Ref. 3]

Ignition of aluminum particles occurs following the breakdown of the surface oxide layer and an induced progressive temperature rise of the aluminum core. Therefore, the most important requirement for ignition is a droplet temperature high enough to melt and retract the oxide layer. Aluminum combustion is delayed even more by the combining of large numbers of particles into large agglomerates (typically 50-200 microns). These droplets require a time of 10-100 ms to complete burning. [Ref. 3]

Aluminum behavior in the combustion process is highly dependant upon the composition of the local atmosphere. The

following equation relates the burning time of an aluminum particle to surrounding gas conditions [Ref. 3]

$$\tau = \frac{kD^n}{\alpha^{0.9}}$$

where τ is the burning time in seconds, D is the initial particle diameter in microns, α is the mole fraction of oxidizing gas, with typical values of k and n of 7.3×10^{-6} and 1.75 respectively. The nature of the combustion zone of an aluminum droplet in a solid propellant rocket motor is depicted in Figure 1.1. The droplet is at approximately 2500 Kelvin, with a residue of molten retracted oxide on the surface and a detached flame envelope. The combustion

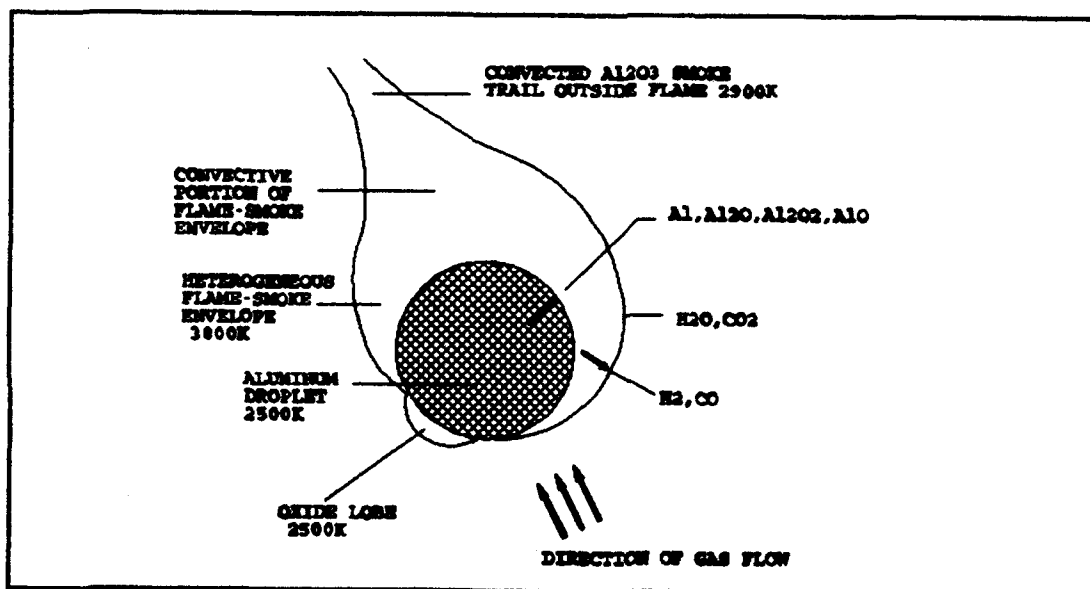


Figure 1.1. Aluminum Droplet Combustion Zone. [Ref. 3]

behavior is constrained by the oxide melting point and the aluminum boiling point. At temperatures below the oxide melting point, more oxide will form on the surface and the flame structure will collapse. Aluminum vapor flows out and reacts to form Al_2O_3 , which is in droplet form (typically greater than 2 microns in diameter). Burning aluminum droplets have oxide on the surface, as well as in the flame envelope. Some of this oxide may be a leftover from the original oxide skin, but additional oxide can form or collect continuously on the burning aluminum droplet. This creates a lobe of retracted oxide which dominates the droplet combustion behavior. [Ref. 3]

The usefulness of boron fuels depends mainly upon particle residence times within the combustion chamber. Boron ignition is complicated by the presence of a liquid layer of boron oxide at a temperature between the melting and boiling points of B_2O_3 . Figure 1.2 is a model of the boron particle ignition taken from King [Ref. 1]. The general model shown accounts for the convective and radiative heat flux to the particle, the reaction of the boron at the boron-boron oxide interface, the evaporation of the B_2O_3 at the gas liquid interface, the diffusion of oxygen through the oxide layer to the boron-boron oxide interface and the reaction of boron oxide liquid with water vapor to form HBO_2 .

Three distinctive characteristics of the boron ignition process were discovered and presented by Macek and Semple

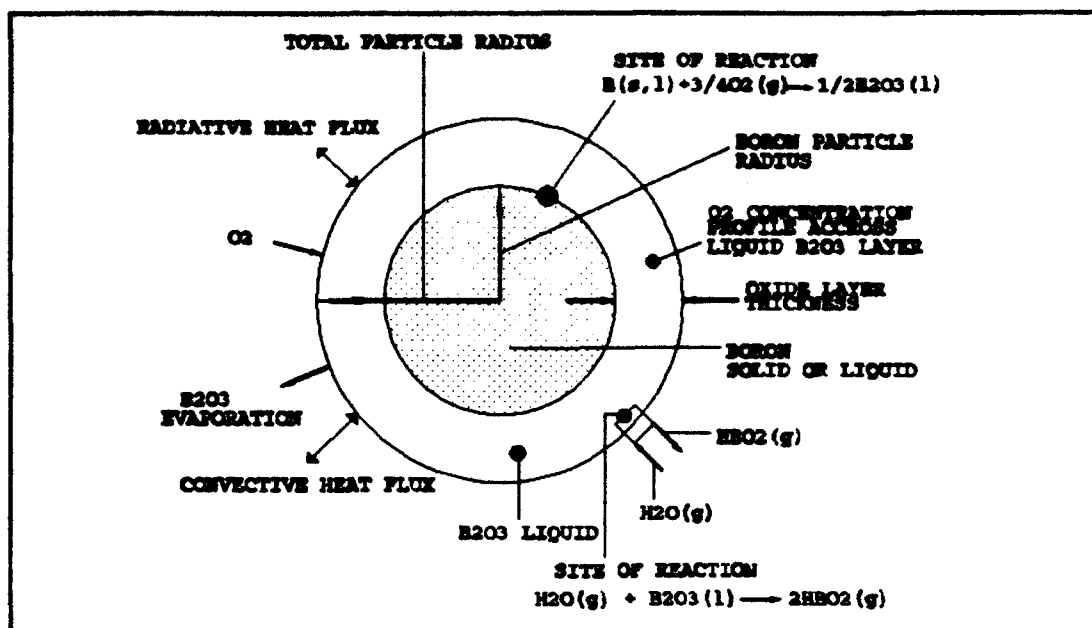


Figure 1.2. Boron Particle Ignition Model [Ref. 1].

[Ref. 4]. The first was that the ignition temperature is always well defined. The second was that the flame structure consists of three zones, a bright central core roughly the diameter of the particle, a wider symmetrical and somewhat less luminous zone and a green envelope zone up to one centimeter wide. The third finding was that boron ignition was a two stage process. In the first stage the particle ignites and burns brightly for a short period of time and then seems to extinguish. Then, in the second stage, the particle reignites and burns more brightly than before in a complete combustion process. Macek and Semple found the average ignition temperature of boron to be $1992 \pm 16^\circ K$ with a water mole fraction equal to zero, and $1869 \pm 24^\circ K$ with a water mole fraction of 0.16-0.21.

Gurevich et. al. [Ref. 5] used oxygen and water vapor as oxidizing mixtures containing 15,30, and 45% by volume with argon and nitrogen. During their experiments they discovered two trends. The first was that ignition temperature decreases with increasing particle size, and the second was that ignition temperature decreases with increasing water vapor content.

In the work done by Macek and Semple [Ref. 4] and Gurevich et. al. [Ref. 5] it was found that the presence of water vapor decreases the ignition temperature and ignition time. These findings show that the presence of water vapor may enhance the removal of the oxide layer in the ignition process [Ref. 6]. Additional characteristics of boron ignition presented were that crystalline boron was significantly harder to ignite than amorphous boron [Ref. 5, 7], and particle self heating was negligible [Ref. 1, 7].

The efficient removal of the B_2O_3 layer is the most important step in ensuring rapid ignition and complete combustion of the boron particle. In the model presented by King [Ref. 1] oxygen dissolves at the outer surface of the oxide layer and then diffuses across the layer to react at the boron surface. It was also noted that second stage ignition takes place when the surrounding or particle temperature exceeds 1900 Kelvin. According to Glassman et. al. [Ref. 8], boron solubility in boron oxide seems to be much higher than oxygen solubility, hence the reaction is likely to take place

at the boron oxide gas interface. A generalization of the oxygen and boron diffusion limiting models may be proposed by a third approach. In this case both boron and oxygen dissolve at the inner and outer surfaces of the oxide layer respectively, and diffuse towards a reaction zone within the layer. Boron does not evaporate during the combustion process like aluminum and hydrocarbon fuels. Once the oxide layer is removed the particle combustion consists of surface reactions rather than gas phase reactions. In spite of the complexity of reaction path, the boron burning process seems to exhibit diffusion controlled behavior and is proportional to the oxygen concentration. [Ref. 9]

A theoretical model for the ignition of titanium coated boron particles in dry air was presented by Gany et. al. [Ref. 2]. Coating of the boron particle with a thin layer of titanium may contribute to speeding up the ignition process in two ways. First, the heating rate of the particle increases due to the reactions of titanium with both boron and oxygen, and second, the mechanical stresses which develop in the TiO_2 and TiB_2 layers may cause their break-up, transforming them to nonprotective films for oxidation of titanium or boron. Under the assumptions made for the development of the theory, the particle ignition occurs in two successive stages as shown in Figure 1.3. During the first stage, the titanium layer is consumed in parallel by the reactions with oxygen and boron which take place simultaneously (Figure 1.3b). After complete

consumption of the titanium layer, oxidation of the boron particle occurs (stage 2, Figure 1.3c). Heating or cooling of the particle results from convective or radiative interactions with the surroundings. Additional heating contribution is due to the exothermic reactions between titanium and oxygen, titanium and boron, as well as boron with oxygen. On the other hand endothermic evaporation processes of titanium oxide and boron oxide extract energy from the particle. [Ref. 2]

During the particle heating process, mechanical stresses appear in the interfaces and may cause break-up of the various layers. The main reasons for this break-up are the difference in thermal expansion coefficients, the difference in densities of the initial substances and final reaction products, and the change in density of the material upon melting. Ignition of the particle is achieved once the titanium is consumed, boron oxide is generated and evaporates at a rate faster than its generation rate, and the boron oxide thickness becomes zero. [Ref. 2]

The effect of titanium thickness from 0.1-1 micron and the surrounding temperature from 1400-2000°K on the ignition time of various diameter particles 5-20 microns was evaluated analytically. The initial thickness of the titanium layer was found to significantly affect the ignition behavior of the particle, with the existence of a critical initial thickness. If the titanium coating thickness is below the critical value the titanium is consumed before the particle reaches a

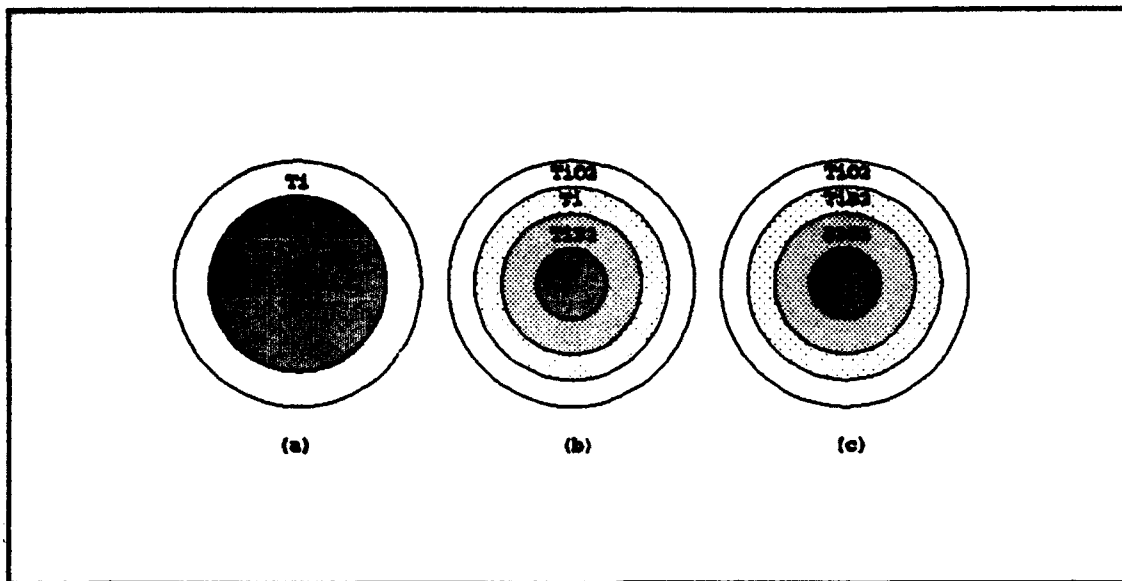


Figure 1.3. Titanium Coated Boron Particle Ignition Model [Ref. 3].

critical temperature necessary for ignition (about 2320°K). Near the critical thickness, the temperature of 2320°K causes the rate of vaporization of boron oxide to be equal to the rate of generation and the particle can ignite (if $t \geq t_{\text{critical}}$) or extinguish (if $t \leq t_{\text{critical}}$). If the initial titanium thickness is above critical, the particle temperature increases up to 2450°K and remains constant until the titanium is consumed and the particle ignites. [Ref. 2]

Initial titanium thickness necessary for particle ignition was found to increase almost linearly with the particle diameter, however the ratio between the titanium mass and the boron mass is almost constant and its maximum value is 7.6%. The critical thickness increases up to 20% of the particle diameter with decreasing ambient temperature to 1400°K, however the critical mass ratio increases much less. The

important conclusion is that the critical titanium mass for most cases is about 8% of the boron mass. In contrast to uncoated boron particles which require ambient temperatures of approximately 2000°K, the titanium coated particles can ignite even at temperatures of 1400°K, due to oxidation of titanium which speeds up the particle heating and promotes the ignition process. [Ref. 2]

In addition to titanium coated particles for enhancing boron combustion, magnesium has been considered due to its ease of ignition. Magnesium generates intense heating and supports ignition and combustion of boron, however the volumetric heat of combustion of magnesium is approximately one third that of boron. Energetic binders (containing oxidizers) such as fluorocarbon polymers, which can initiate boron particle heating and increase burning rate, should be used in the minimum portions necessary to prevent significant reductions in the specific impulse. Other binders, copolymers such as BAMO/NMMO, have been shown to produce energetic surface reactions, lower surface temperatures (than HTPB binders), and better dispersion of boron particles into the gas phase. Increasing oxygen content has also been shown to increase the burning rate of boron. [Ref. 9]

Considerable theoretical modeling has been accomplished for the combustion of various metallic materials, but few data have been reported to validate these models for the burning rates as a function of the surrounding gas composition and

pressure. This is especially true for the bimetallic and coated particles and for particles placed in environments containing combustion enhancers. Therefore, in the present study, an apparatus was designed to obtain data for the ignition characteristics (particle temperature) and burning rates of metallic (or liquid) particles. The end objective for the apparatus was to determine methods/environments which could decrease ignition time and increase burning rate.

A key diagnostic in the experiment was the measurement of particle size. The sizes of large particles ($d > 5\lambda$) can be determined from the intensity of scattered light [Ref. 10]. A schematic of a typical experimental setup for measuring forward scattered light is shown in Figure 1.4. For particles larger than 10 microns the light in the center lobe

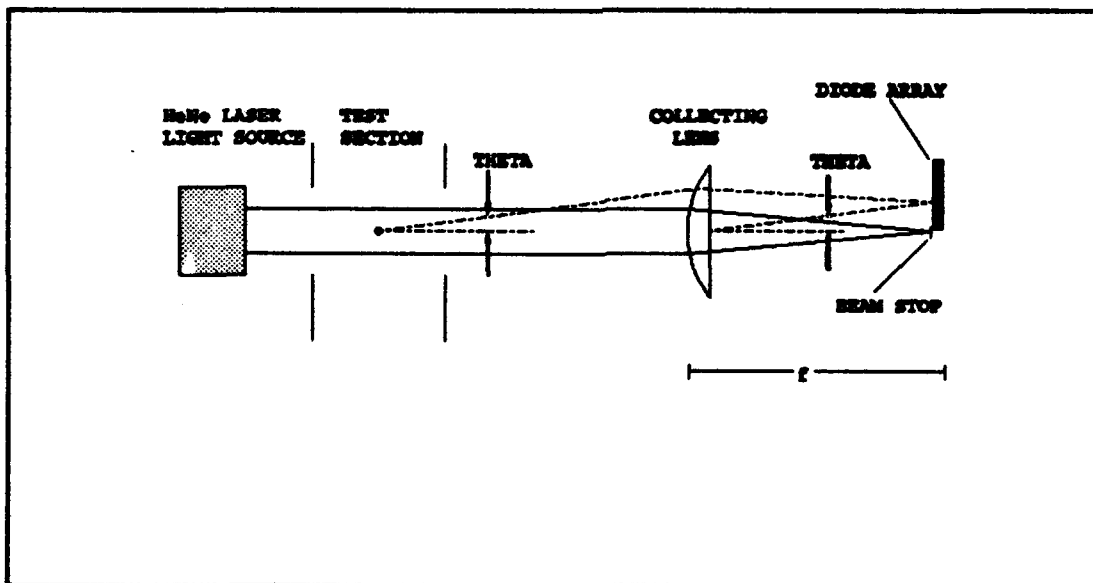


Figure 1.4. Measurement of Forward Scattered Light.

of scattered light is primarily due to Fraunhofer diffraction. Buchele [Ref. 11] is one example of the application of Fraunhofer diffraction for sizing particles. Any light scattered by a particle in the test section will be focused on a diode array at the focal plane of the collecting (Fourier transform) lens. The angle from the center of the collecting lens to the point at which the scattered light is focused is the same angle(θ) that the light is scattered. The curve for the distribution of the scattered light is given by

$$\frac{I(\theta)}{I(0)} = \left[\frac{2J_1(\alpha \sin(\theta))}{\alpha \sin(\theta)} \right]^2$$

where $I(\theta)$ is the intensity of light scattered at an angle θ , $I(0)$ is the forward scatter light intensity at $\theta=0$, $\alpha = \pi D/\lambda$ (the particle size parameter), θ is the light scattering angle, and J_1 is the first order Bessel function. For angles less than 20 degrees, $\sin(\theta)$ may be approximated by θ in radians. For large particles the highest intensity of scattered light is located in the center lobe at an angle of zero degrees. Unfortunately, the intensity of scattered light in this lobe cannot be measured due to the presence of transmitted light at this angle. This phenomena gives rise to the need for a beam stop as shown in Figure 1.4. The size of the beam stop limits the upper particle size that can be

measured since the larger particles scatter more light at the smaller angles. [Ref. 11]

The apparatus that was designed and built, and the ignition and diagnostic systems that were used, are described in the following section.

II. EXPERIMENTAL SETUP

A. COMBUSTION BOMB

The combustion bomb designed and fabricated for use in this investigation consisted of three sections: the top, main body, and the bottom. A schematic of the bomb is displayed in Figure 2.1.

The top portion consisted of a stainless steel plate with a 1/8-inch NPT fitting connected to a vacuum pump. A piece of 50 micron inner diameter, 375 micron outer diameter quartz tubing was epoxied into a ceramic tube affixed in the NPT fitting to hold a single particle for ignition. The bottom portion of the apparatus consisted of a port for the introduction of gasflow and a piece of 1/4-inch thick sintered bronze to ensure uniform flow.

The main body of the apparatus contained two one-inch diameter synthetic fused silica windows on each of the opposing wide sides of the body. The narrow sides of the body had two 1/2-inch diameter windows each. The top window on each of the narrow sides was made out of zinc selenide for use specifically with a carbon dioxide laser. The bottom windows on each narrow side were synthetic fused silica. The main body also contained a port to measure static pressure and an

exhaust valve. The exhaust valve could be used to provide either a steady gas flow through the bomb or a stagnant atmosphere.

B. IGNITION SYSTEM

The ignition system for this investigation was to be a 20 watt CO₂ laser. However, the laser received on loan was a Laakman Electro-Optics RF 44 Carbon Dioxide Waveguide Laser System with a power of only 5 watts. The laser had a wavelength of 10.6 microns and a beam diameter of 1.7 mm. A zinc selenide plano convex focusing lens and a silver coated silicon concave spherical mirror were also used (Figure 2.2).

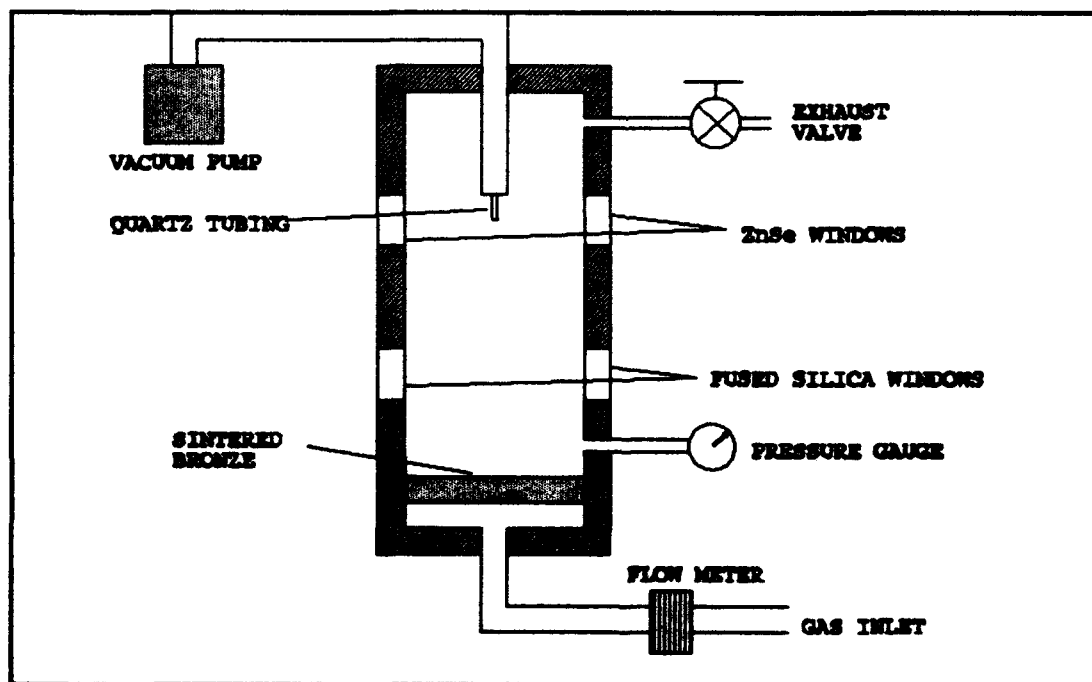


Figure 2.1. Schematic of Combustion Bomb.

The lens had a focal length of 63.5 mm and focused the beam down to a minimum waist diameter of 0.17 mm. The mirror was placed opposite the lens at a distance of 63.5 mm (radius of curvature of the mirror) from the focal point of the lens (center of the combustion bomb) to provide heating of the particle from both sides.

C. DATA ACQUISITION SYSTEM

1. AGEMA Thermal Imaging System

The AGEMA Thermovision 800 consisted of a scanner, control unit, monitor, and a power supply.

The scanner unit consisted of a fixed lens, an electro-optical scanning mechanism, and infrared detector, video interface and control electronics and microprocessor for data output. The detector was mercury cadmium telluride mounted on a sapphire substrate, and was sensitive between two and five microns. The scanner can be used with any one of five different standard lenses with fields of view varying from 2.5 to 40 degrees. However, the scanner was used in the fixed lens configuration for this investigation. The fixed lens is used for macro work with a focal length of 20 mm and a field of view of 13 mm by 13 mm displayed on the screen.

2. Particle Sizing System

The light source used for demonstrating the Fraunhofer diffraction technique was a 15 milli-watt Spectra-Physics helium-neon laser. The laser beam was passed through a

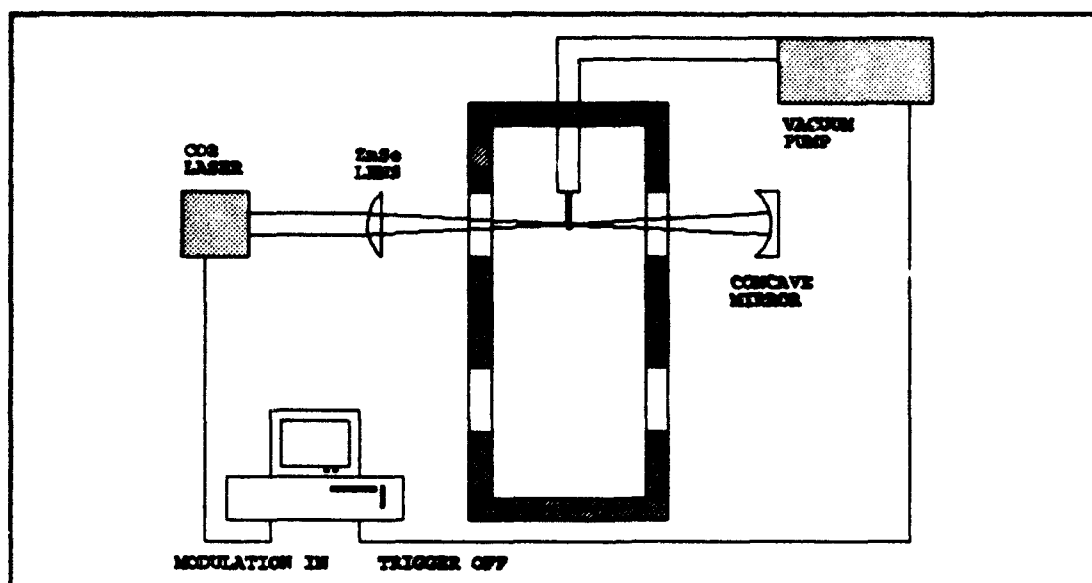


Figure 2.2. Ignition System.

spatial filter/beam expander to create a larger diameter beam. This ensured full coverage through the chamber cavity cross section and also increased the particle residence time within the beam to permit more data to be collected. The scattered laser light from the particle(s) was collected by a plano-convex lens and focused on a linear diode array 200 mm away. A beam stop was located at the focal point of the lens to block the main transmitted beam. The clock rate for the EG & G Reticon 1024G diode array and RC100B Board Alignment operated at 1 Mhz for this investigation, to match the speed of the data acquisition system. Figure 2.3 is a schematic of the data acquisition system.

3. Data Acquisition Board and Software

The Keithley Metrabyte DAS-58 is a very high speed A/D data acquisition board that is capable of sampling at a rate

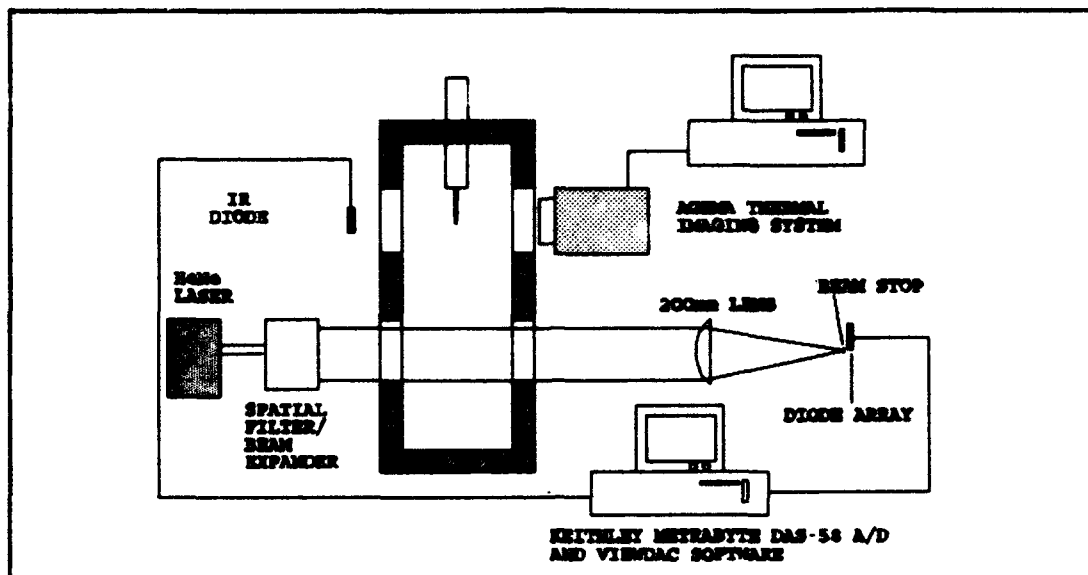


Figure 2.3. Data Acquisition System.

of one megasample per second and has an onboard storage capacity of one million samples. It is an eight channel analog input board with simultaneous sample and hold. The software package used to control the data acquisition board and the apparatus was VIEWDAC. This software provided data acquisition and control, analysis, and graphical data presentation.

4. Data Reduction

The EG & G Reticon 1024G internal clock was output to and used by the DAS-58 data acquisition board to ensure synchronization between the acquisition system and the diode array. When operated at 1Mhz, the system could sweep the diode array eight times while the particle was passing through the expanded beam. The diodes were spaced at 25 microns center-to-center, which corresponded to a scattering angle of

0.0072 degrees per diode with the 200 mm focal length lens. This provided a maximum scatter angle of 7.4 degrees. These eight sweeps of the diode could be averaged and simultaneously plotted with theoretical curves. From these curves the best fit could be selected and the particle size determined. Several theoretical curves are shown for various particle sizes in Figure 2.4.

An IR diode was positioned outside an upper window to sense the ignition and trigger data acquisition. The burning time could then be obtained from the difference between the start time and the time at which the scattered light was sensed (the total number of sweeps and the clock rate). Then the burning rate could be determined from the initial and final particle sizes and the burning time.

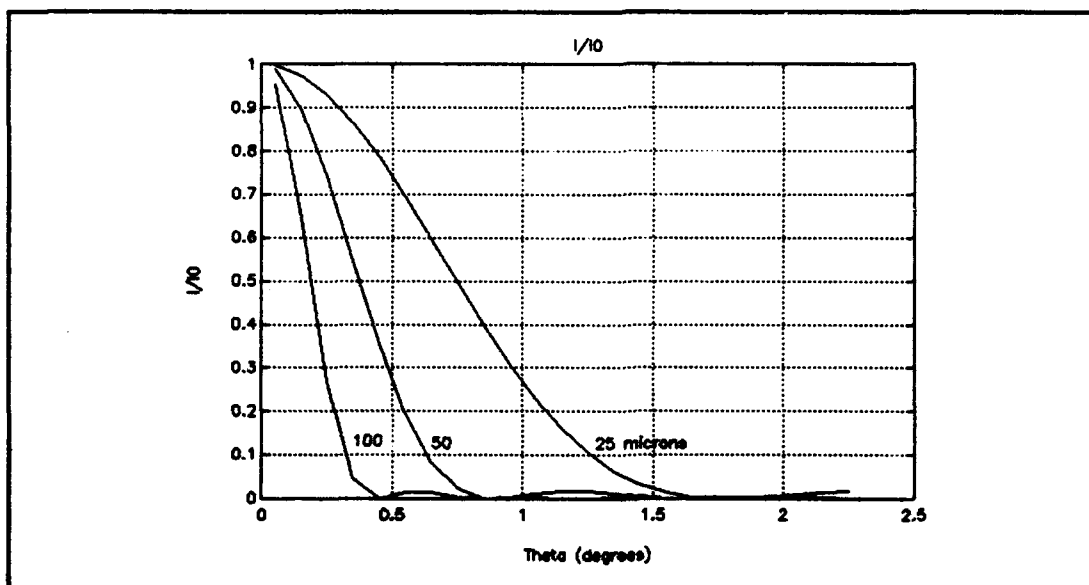


Figure 2.4. Theoretical Light Scattering Profiles.

III. RESULTS AND DISCUSSION

A. IGNITION

Initially a 95 micron aluminum particle was held by the vacuum system and dropped through the focused CO₂ laser beam. This was unsuccessful. As expected, the 5-watt laser was not powerful enough to heat the particle to the ignition temperature while it was transiting the beam. A solution to this problem was attempted by holding the particle within the focused beam for a short period of time (5-10 msec). This also was unsuccessful. The outside diameter of the quartz tubing was 375 microns (roughly four times the particle diameter) which made distinguishing between the particle and the tubing extremely difficult with the IR camera. Another difficulty associated with holding the particle in the beam was that the beam was also incident upon the quartz tubing. The energy impinging upon the tubing was high enough to melt the tip of the tubing and seal off the vacuum. Also, the particle was stuck to the end of the tubing.

To detect the ignition event the AGEMA thermal imaging system was operated at its maximum rate of 25 frames per second. During this time a particle beginning to fall from just above the focused laser beam would be in view of the AGEMA system for approximately one frame. To determine if the

heated particles could be detected, several particles were dropped through a 700 micron inside-diameter quartz tube and into the focused laser beam. Figure 3.1 shows that the thermal image could be captured using the 25fps framing rate. The particle(s) is the oval shaped spot in the upper left center portion of the figure. The oval shape was probably due to several particles being stuck together.

The current ignition system was inadequate. The ideal solution to the problem is to obtain a more powerful CO₂ laser to ignite the particles as they pass through the beam. Macek and Semple [Ref. 12] have proven that an 80 watt laser is powerful enough to ignite falling particles of B₄C. The required laser power can be estimated using the following equation

$$P\alpha = \rho_p \text{Vol}_p C_v \Delta T$$

where P is the laser power, α is the particle absorptivity, ρ_p is the particle density, Vol_p is the particle volume, C_v is the specific heat and ΔT is the difference between the initial particle temperature and the temperature required for ignition. Assuming an absorptivity of 0.5, a beam diameter of one millimeter and a particle diameter of 100 microns, the power required to raise the particle from room temperature to

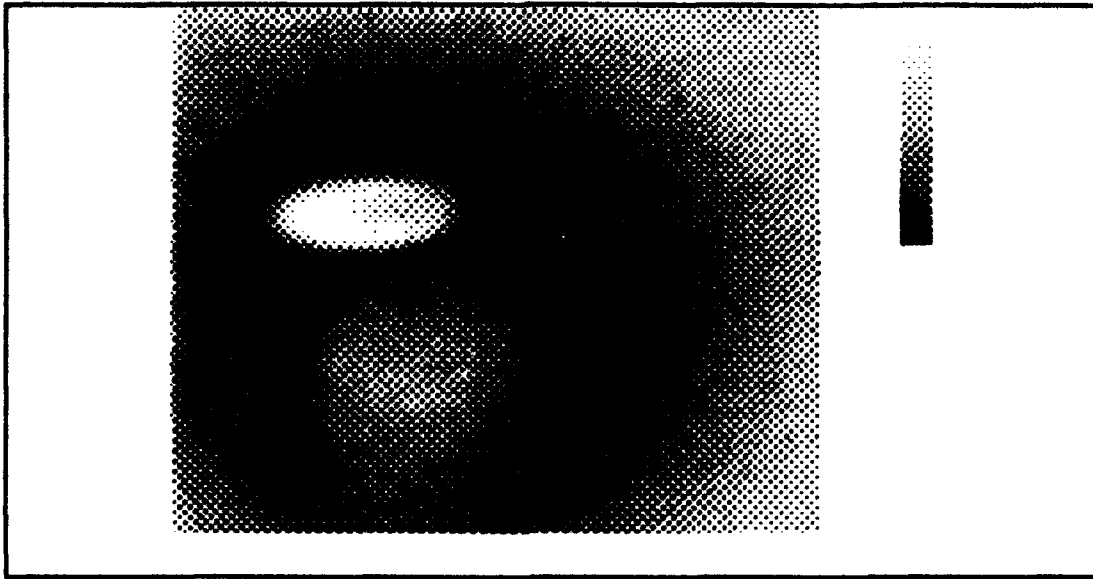


Figure 3.1. Thermal Image of a Falling Particle.

ignition temperature for boron and aluminum was found to be 105 and 43 watts respectively. For a beam diameter of 0.5 mm, the power drops to 37 watts for boron and 15 watts for aluminum.

B. PARTICLE SIZING SYSTEM

The clock signal output from the EG & G 1024G linear diode array was initially unable to be used by the DAS-58 data acquisition board and VIEWDAC software. This was apparently due to a malfunctioning DAS-58 board. After a repaired board was received the system functioned properly. The expanded 15 mw He-Ne laser beam used to check out the particle sizing system was known to not provide enough scattered intensity from one particle (< 25 micron diameter) to be detected by the diode array. The expanded beam was required to increase the

probability of the particle passing through the beam after falling from the top of the combustion bomb. Therefore, to perform a system check, very low concentrations of particles in water and in air were measured in the unexpanded beam. Successful measurements were obtained by multiple sweeps of the diode array.

Table 2 shows the laser (beam diameter=1mm) power required for measuring the intensity of scattered light at approximately the center of the first Airy diffraction pattern, for various sizes of single particles, using the diode array. These values are based upon a scattered light intensity of twice the noise level (approximately 7.5 $\mu\text{watts/cm}^2$) of the EG & G 1024G array.

TABLE 2. REQUIRED POWER FOR SINGLE PARTICLE SCATTERING.

DIAMETER (microns)	POWER (Watts)	SCATTERING ANGLE (degrees)
5	72	14.5
15	1	1.5
20	0.28	1.2
25	0.012	0.9

The largest particle sizes for commercially available metals such as Al, B, B₄C is approximately 100 microns. The approximate burn time for a 100 micron initial diameter, 20 micron final diameter B₄C particle is 190 msec [Ref. 12]. This corresponds to a required free-falling distance of 6-7 inches. The problem with the initially conceived idea of

measuring scattered light from a single particle using a laser of reasonable power (< 4 watts) was to get a burning particle to fall straight down the bomb and pass through a one millimeter diameter laser beam. Two practical solutions are possible. The first is to incorporate an acoustic vibrator/particle feeder similar to the one used by Macek and Semple [Ref. 12] to allow multiple particles to pass through the CO_2 laser beam, ignite and then pass through the diagnostic laser beam. The presence of multiple particles would increase the scattered light intensity. This would decrease the power necessary for detection, and increase the probability of detection through the use of an expanded beam. The second solution is to incorporate a Phase Doppler Particle Analyzer (PDPA) for particle size measurement. The PDPA measures particle size from the phase shift produced by particles passing through crossed laser beams, rather than from the magnitude of intensity of scattered light. The PDPA can measure particle sizes from 0.5 to 1000 microns with a dynamic range, with any individual set of optics, of 50. Thus, particles from 0.5-25 microns could be measured. However, the PDPA sample volume is small and will require many particles (10-100's) to ensure a statistically valid sample. Figure 3.2 is a schematic of the proposed modifications to the existing combustion bomb which would permit the original objectives of this investigation to be accomplished.

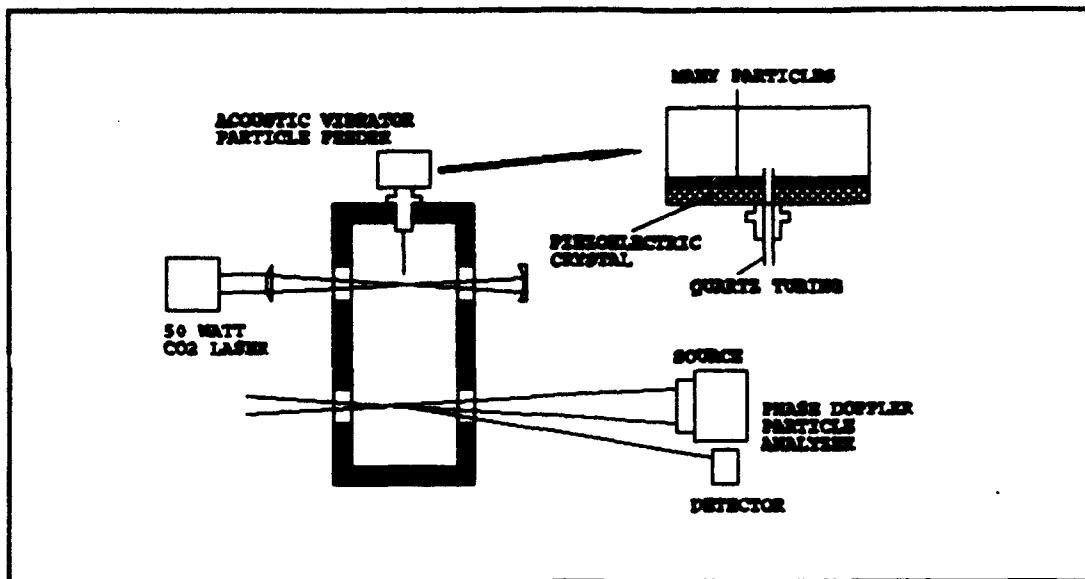


Figure 3.2. Recommended Modifications to Apparatus.

IV. CONCLUSIONS

An apparatus was designed and fabricated for measurement of ignition and burning characteristics of metallic particles or liquid fuel droplets. Modifications to the apparatus must be incorporated before the apparatus can be used for its intended purpose. These modifications include a more powerful CO₂ laser for particle ignition, an acoustic vibrator/particle feeder to provide multiple-particle burning and a Phase Doppler Particle Analyzer for measuring the final particle diameters.

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